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Investigation of the critical shear stress for single crystals of metallic solid solutions. I

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With 16 figures in the text

Introduction

The aim of the present work is to find the resolved critical shear stress τ_0 for single crystals of dilute solid solutions of copper, silver or gold as solvents and arbitrary other elements as solutes; the quantity τ_0 is defined as the stress resolved on the slip plane and in the slip direction at which a crystal will begin to flow at an appreciable rate.

Measurements on this subject for alloys with face centered cubic structure have previously been made by v. Göler and Sachs (1) (Cu-Zn), Sachs and Weerts (2) (Ag-Au) and Osswald (3) (Cu-Ni).

In this paper a description is given of measuring methods and the methods of producing single crystals and also some results obtained with the apparatus developed.

1. Production of specimens

The components of the alloys were first melted together in evacuated sealed-off silica tubes and homogenized in the molten state with the aid of high frequency currents (in some cases by mechanical shaking). The polycrystalline castings obtained were transferred to a graphite crucible for remelting and growing to single crystals in the furnace shown schematically in Fig. 1. The method for producing the crystals is that described by Bridgman (4). During the solidification of the melt, the furnace is moved upwards at a rate of about 2 cm/hour by an electric motor. As the conical bottom of the crucible (Fig. 2) will first leave the hottest zone of the furnace, solidification will begin at the peak of the cone. The solidification front advances slowly upwards through the melt, which results in the growth of a single crystal.

Fig. 3 gives a total view of the arrangements for the crystal production.

After cooling to room temperature the crucible was cracked and the specimen freed. This operation had to be done very carefully in order to avoid disturbing the crystal. A groove was cut in the crucible which was then split open. The specimen was etched (for the Cu alloys: 1 part HCl, 1 part H_2O; mixture saturated with $FeCl_3 . 6\,H_2O$). Direct observation of the selective re-

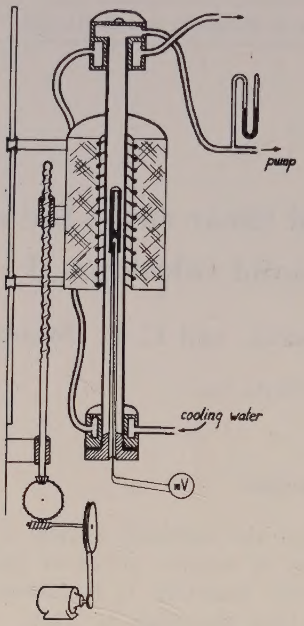

Fig. 1.

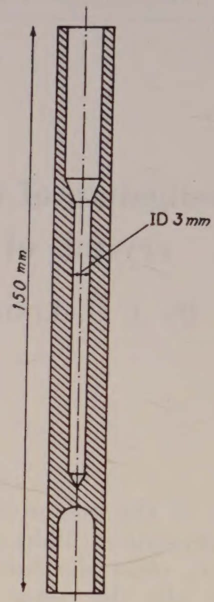

Fig. 2.

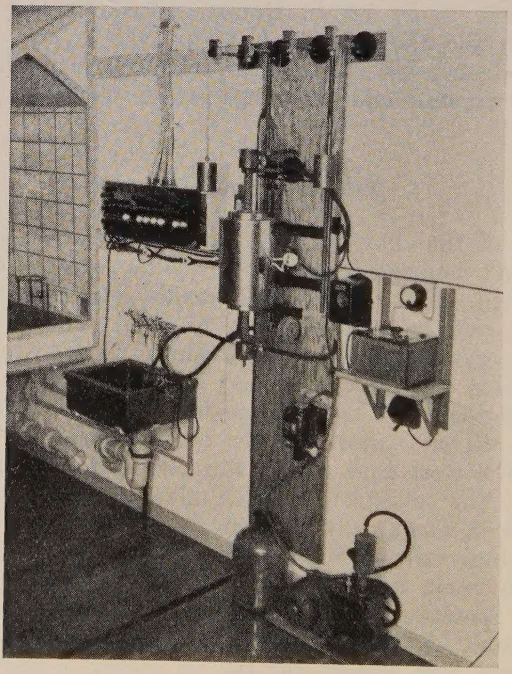

Fig. 3.

flection of light showed then whether or not each was a single crystal. The accepted crystals were heated at high temperature (about 1000° C) during 10 to 15 hours in order to remove segregations produced during solidification.

2. X-ray technique

The homogenized single crystals were examined by X-rays in order to check homogeneity and to find the orientation of the crystal axes in relation to the axis of the rod. Fig. 4 shows the application of the crystal rod in the X-ray camera, which was constructed for cylindrical film. The X-ray tube was of the sealed-off type with an iron target. The time of exposure was about 20 minutes, with a current of 15 mA and a voltage of 30 kV.

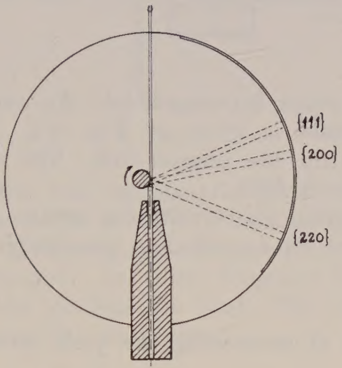

Fig. 4.

The photographs obtained usually showed the sharp patterns characteristic for undeformed crystals (Fig. 5). In some cases however the patterns showed a broadening, indicating that the crystal had been slightly deformed plastically in handling. Only specimens that gave sharp patterns in the X-ray photo-

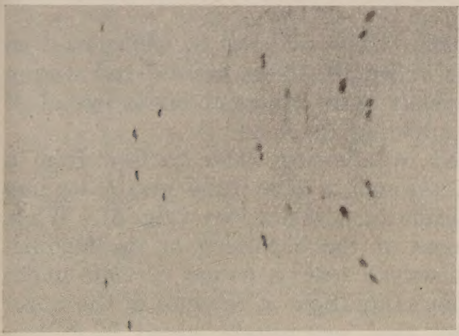

Fig. 5 a.

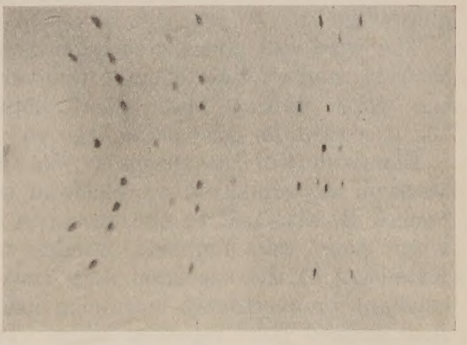

Fig. 5 b.

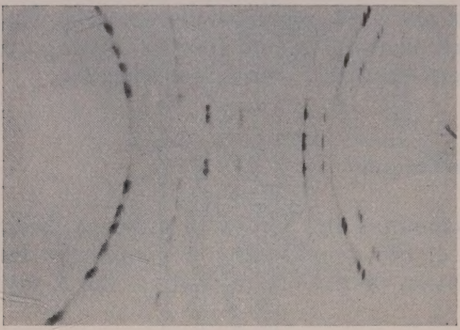

Fig. 6 a.

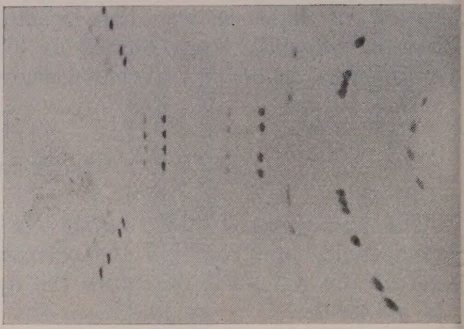

Fig. 6 b.

graphs were used for further investigations. An example of an X-ray photograph of deformed crystals is given in Fig. 6 a. It is obtained from the specimens of Fig. 6 b after the tension test. The elongation of the specimen after plastic flow was about 0.02 %.

The method of evaluating the orientation of the axes of the single crystals from the photographs will be described in paragraph 4.

3. The method of measuring the yield stress of the crystals

The stress-strain curve for the crystal rods was determined with an electric resistance method.

Trials were first made with strain gauges of commercial type consisting of thin metal wires embedded in a plastic material which was fastened directly onto the crystals. Some measurements were also made with this arrangement. However there was reason to doubt the results obtained owing to the fact that the gauges influence the yield point, causing too high values of this quantity. After various experiments the following arrangement was finally adopted which seems to be free from systematic sources of error. A thin constantan wire (0.025 mm diameter) was attached to the surface of the rods. At one end the wire was soldered directly to the crystal; at the other end it was attached by glue (Fig. 7).

The wire was given a suitable elastic strain when soldered to the crystal so that it might follow exactly the variation of length of the crystal rod during the whole tension test. Small copper eyelets were soldered on each end of the specimen for suspending the rod and applying the load.

Elongation of the specimen was obtained by allowing water to flow from a reservoir A, supported on platform scales, through a glass tube into a loading bucket B, attached to the specimen by means of a metal wire (Fig. 8). When large loads were required, weights were used at the beginning of the loading. Extension of the specimen may thus be observed over a period of time under constant or constantly increasing load. The calibration of the rate of the water flow showed good reproducibility. The rate used was usually about 50 g/minute. The electrical circuit was a Wheatstone bridge shown in Fig. 8. The

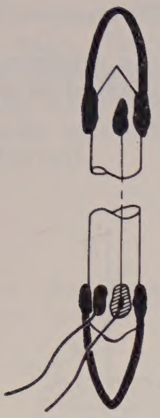

Fig. 7.

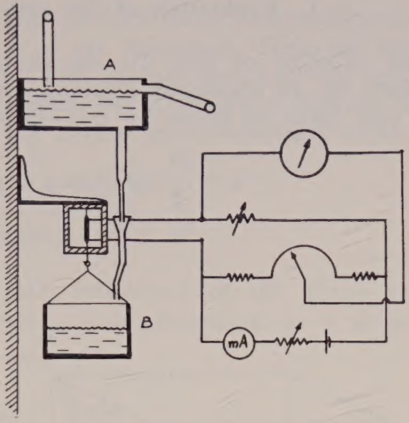

Fig. 8.

sensitivity of the apparatus was sufficient to give a galvanometer deflection of 1 mm for a strain of the order of 10^{-6}. The simultaneous readings of load and galvanometer deflection give a stress-strain curve. In Fig. 9 examples are given of the curves obtained. In these diagrams the ordinate gives the load and also the time of the reading the load. The yield stress σ_0 used in the calculation of τ_0 is indicated in the figures.

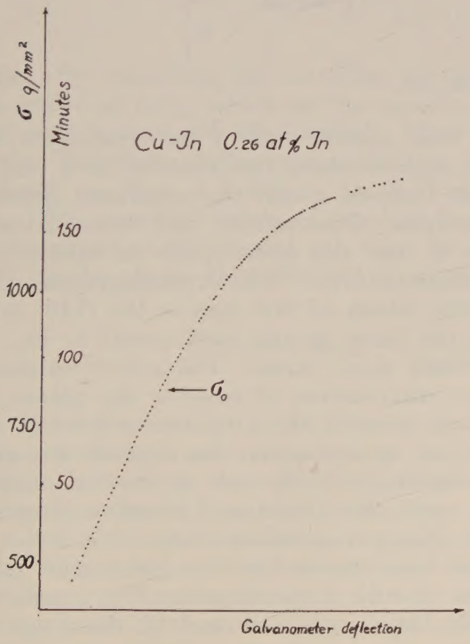

Fig. 9 a.

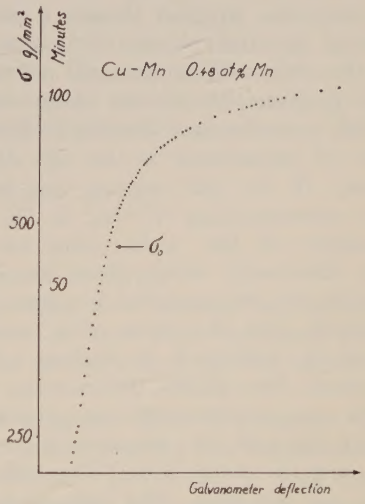

Fig. 9 b.

4. Evaluation of the critical shear stress τ_0

With the symbols of Fig. 10, the shear stress τ in an arbitrary crystallographic plane of an elastically strained single crystal, resolved in a given direction in this plane, is given by the expression

$$\tau = \frac{P}{A} \cos\lambda \cos\phi = \sigma \cos\lambda \cos\phi \tag{1}$$

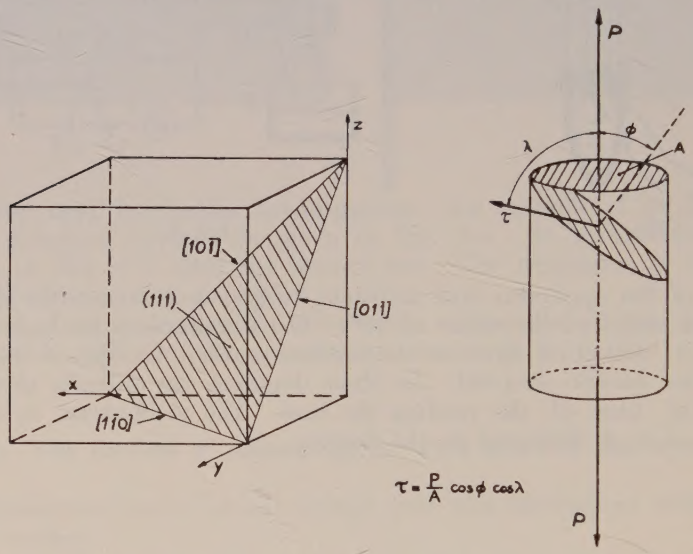

Fig. 10.

where P is the applied force, A the cross section area of the crystal rod, and σ thus the applied stress. ϕ is the angle between the direction of σ and the normal to the plane. λ is the angle between σ and the considered direction in the plane. The essential crystallographic plane coming into consideration in the problem of critical shear stress is the slip plane (plane of easiest slip), which for the face centered cubic metals is (111). The crystallographic direction of importance is the slip direction, which in this case is the $[1\bar{1}0]$ directions. If we put $\sigma = \sigma_0$ (σ_0 being the stress at the yield point) in Eq. (1), the corresponding $\tau = \tau_0$ is the critical shear stress. The active translation elements at the yield point are that combination of possible slip planes and slip directions which gives the greatest value of the product $\cos\lambda \cos\phi$. According to an empirical law the quantity τ_0 is constant for crystals of a given material, but the value of τ_0 varies slightly with the rate at which the applied stress is increased in testing. For very slow rates and sensitive measuring methods, the plastic deformation first observed is called creep.

In our experiments σ_0 is obtained from the load at the yield point (paragraph 3) and by measuring the area A with a micrometer. The accuracy of the area is about 2 %. This relatively large error is caused by the rough surface of the rods. The rate of increase of σ used in the experiments is about 5 g mm^{-2} $\mathrm{min.}^{-1}$ (Fig. 9).

The evaluation of $\cos \lambda \cos \phi$ of Eq. (1) from x-ray photographs can be made in various ways. The method chosen is the following. With the camera used, which was constructed for cylindrical film (radius $R = 2.88$ cm) and the symbols of Fig. 11, the following expression for the angle ϱ between the axis of rotation (axis of crystal rod) and the normal of an arbitrary crystallographic plane is obtained,

$$\cos \varrho = \frac{m}{2 \sin \vartheta \sqrt{R^2 + m^2}}$$

where ϑ is the Bragg angle for the reflections of the plane and $2m$ is the distance between two symmetrical spots belonging to a given group of planes. By measuring the m values for the (200) group we obtain three values, a, b, and c of $\cos \varrho$, which are the direction cosines of the rod axis related to the cubic axis system of the crystal. Here $a^2 + b^2 + c^2 = 1$. By choosing the positive directions of the axes so that a, b, and c are all positive, a sequence is chosen so that $a \leq b \leq c$. (a, b, and c being related to the x, y, and z axes. The possible values of $\cos \lambda$ and $\cos \phi$ for the various slip planes and slip directions of the crystal can then be expressed in the quantities a, b, and c. It can be shown that the maximum value of the product $\cos \lambda \cos \phi$ is

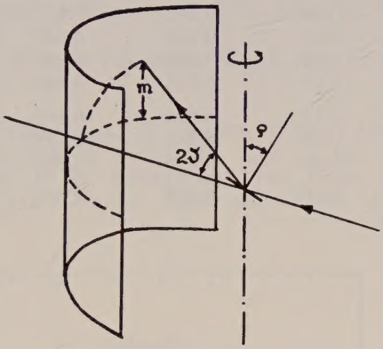

Fig. 11.

$$(\cos \lambda \cos \phi)_{\max} = \frac{1}{\sqrt{6}} (a + c)(b + c - a).$$

Thus this value is used in Eq. (1) in the calculation of τ_0.

The calculations and measurements can, of course, also be made on the basis of other reflections than (200). In fact we have always used combined measurements on the (111) and (200) reflections in our evaluation of τ_0, thus checking the values obtained from different groups.

The homogeneity of the crystal rods was investigated after the tension test by rolling and drawing the rods to long wires and measuring the resistance on different parts of them in distances of about 10 cm. The result of such measurements showed that the degree of homogeneity varied from crystal to crystal. For example the sample with 0.48 at % Mn was within the error of measurement quite homogeneous, but for the other crystals a variation in resistance of 1–2 % from the mean value was found.

In Table 1 the measured values of the yield stress, cos ϕ and cos λ and τ_0 for Cu and some Cu-Mn, Cu-Sn and Cu-In alloys are put together. In Fig. 12 τ_0 is plotted against the concentration c of the solutes. The curves obtained are straight lines within the accuracy of the measurements. Considering the open scale for the concentration axis, it is reasonable to assume that the scattering of the points is mainly due to the uncertainty in c. A reasonable error in c of 0.02–0.03 % Mn or Sn could explain the deviation of the points from the straight lines of the figure. The concentration of the samples has been calculated or controlled from the resistivity values obtained in these measurements, taking the mean value for the whole sample. The values of the increase in atomic resistivity of different solutes, given by LINDE (5), have been used in these calculations.

Table 1

Alloy	c at %	cos ϕ	cos λ	τ_0 g mm^{-2}
Cu		0.692	0.717	53
Cu-Mn	0.48	0.657	0.717	220
	0.52	0.746	0.645	240
	0.61	0.390	0.836	230
	0.70	0.551	0.800	290
Cu-Sn	0.12	0.593	0.802	220
	0.20	0.683	0.718	320
	0.30	0.535	0.834	490
Cu-In	0.26	0.658	0.660	350

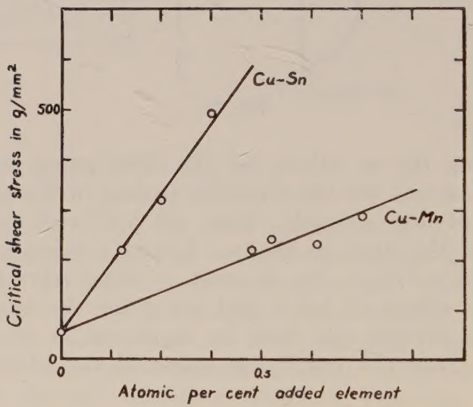

Fig. 12.

SUMMARY

Single crystals have been made from melts of copper and dilute copper alloys. Experimental arrangements have been developed for the determination of the critical shear stress of the alloys, using an electric method in which stress was measured by means of a thin resistance wire (strain gauge) fastened to the crystal. The crystallographic orientation of the sample rods was determined by X-rays. Preliminary results show that the critical shear stress increases strongly with the concentration of solute elements. For solutions of Mn in copper the increase in shear stress per atomic percent is much lower than for Sn in copper.

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